Swami Ramanand Teerth Marathwada University, Nanded, Maharashtra, India

(UGC recognized, NAAC Re-accrediated 'B++' grade state university)

"Synthesis and Characterization of Metallic and Non-metallic Nanoparticles using Green Methods"

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Introduction to proposed research topic

Nanotechnology is a well established area of research now days and many researchers across the world are engaged in continuous research in this area. The decrease in particle size leads to increase in surface area which aids useful properties to the materials. Nanoparticles have a high specific surface area and unique physicochemical characteristics which has motivated researchers to work in this area. There are several types of nanoparticles which are differentiated based on their size, shape, composition and their functionalities. It mainly deals with synthesis of nanoparticles using various methods and finding their applications in diverse fields of life. The nanoparticles range from 0 to 100 nm range in at least one of the dimensions and are having greater surface to volume ratio, unique optical, electrical, thermal, catalytic and antimicrobial properties [1]. The nanoparticles with controlled size and composition are of fundamental and technological interest owing their ability to provide solution to various technological and environmental challenges such as solar energy conversion, catalysis, medicine and water treatment etc. Till date variety of metallic and non-metallic nanoparticles including metal sulphides, metal-oxides, metallic, bimetallic, hybrid nanocomposite, nanocatalysts have been synthesized and studied intensively using various characterization methods.

Nanoparticles can be easily prepared using various physical and chemical processes such as mechanical method, pulse laser ablation, pulsed wire discharge, chemical vapor deposition, laser pyrolysis, ionized cluster beam deposition, sol-gel, sonochemical synthesis, co-precipitation, inert gas condensation, hydrothermal synthesis method etc [2]. These conventional methods suffer from limitations such as use of hazardous chemicals and solvents, tedious process and high cost factor.

To overcome this several biological or green methods are employed in the synthesis of nanoparticles. These methods include use of bacteria, fungi, yeast and extracts of various fruits and various parts of plants for the synthesis of nanoparticles. These methods are simple, eco-friendly, reliable, biocompatible, economic and attracted attention of researchers around the world [3]. The various biomolecules such as alkaloids, flavonoids, carbohydrates, glycosides, saponins, tannins, steroids, terpenoids and proteins present in biological extracts are reported to assist the synthesis of nanoparticles.

Purpose of carrying out proposed research work

The main focus of researchers is on the synthesis of metal and metal oxide nanoparticles such as silver, gold, copper, palladium, platinum, bismuth, titanium oxide, zinc oxide, magnesium oxide etc. From literature survey it is clear that, approximately 29% of total research publications in this field of nanotechnology are devoted to metal nanoparticles synthesis. The non-metalic elements such as carbon, silicon, phosphorus and sulfur are also not far behind in this race, are synthesized and reported to have various applications.

The problems faced by researchers in exploring physical and chemical methods for the synthesis of nanoparticles have forced them to develop eco-friendly or green processes of nanoparticle synthesis. This has led to emergence of new branch in this area namely biomediated or biological (Biogenic) or green synthesis of nanoparticles, which is a greener alternative and hence is more effective. Although this is a well established research area and many research publications are available in this area, there is enough scope to work. The availability of huge amount of biological materials in nature, which can be explored for the synthesis of nanoparticles, has motivated us to undertake synthesis of metallic and non-metallic nanoparticles using green methods and thus increases demand for it.

Survey or review of literature and development in the relevant subject

The involvement of many researchers in this area has led to huge amount of publications in this area. As it is not possible to provide complete account of research publications here, few representative publications are mentioned. The review of literature is presented to convince the importance of synthesis of nanoparticles by using green methods to the readers and familiarize them with various characterization methods adopted.

Khan et al [4] have reported green synthesis of silver nanoparticles using *coriandrum sativum* leaf extract. The UV-Visible spectrometer was used to monitor the reduction of Ag^{+} ions and formation of AgNPs in medium. The XRD and SEM methods were used to investigate the morphology of prepared AgNPs. The peaks in XRD pattern indicated face centered cubic (FCC) form of metallic silver.

Amini et al [5] have reported green synthesis of silver nanoparticles using *Avena Sativa* leaf extract at different temperatures (30, 60, 90^{o}C) and $AgNO_3$ concentrations (1, 2 and 4 mM). The morphology and size of the nanoparticles were determined using Scanning electron

micrcoscopy (SEM) and Dynamic light scattering (DLS) techniques. Based on the results the synthesized nanoparticles were proposed to have particle size in the range from 60-100 nm.

Awwad et al [6] have reported green synthesis of sulfur nanoparticles using *Rosamarinus officinalis* leaves extract and sodium thiosulfate pentahydrate at room temperature. The synthesized sulfur nanoparticles were characterized using UV-Visible spectroscopy, FT-IR, SEM, TEM and XRD techniques. The obtained results indicated that the synthesized nanoparticles were of particle size of 40 nm. The synthesized nanoparticles were also evaluated for their nematicidal activity against *Meloidogyne javanica.*

Awwad et al [7], in another research paper, have reported green synthesis of sulfur nanoparticles using *Punica granatum peels* aqueous extract at room temperature. The resulting sulfur nanoparticles were characterized by XRD, FT-IR, SEM and EDS techniques. Based on the results obtained the sulfur nanoparticles were proposed to exhibit high purity, spherical shape with average particle size of 50 nm. The synthesized sulfur nanoparticles were also evaluated for their effects on the growth of tomato by foliar spray application method.

Khairan et al [8] have reported green synthesis of sulphur nanoparticles using sodium thiosulphate pentahydrate and in presence and absence of aqueous garlic extract *(Allium sativum).* The resulting sulphur nanoparticles were examined using FT-IR, XRD and SEM analysis. Based on the results obtained the nanoparticles were proposed to have particle size of 55.613 and 70.908 nm respectively. The synthesized nanoparticles were tested for their antifungal activities using disc diffusion method.

Tsuzuki et al [9] have reported green synthesis of sulfur nanoparticles using *F. benghalensis* leaf extract. The leaf extract was proposed to act as a reducing and capping agent. The synthesized sulfur nanoparticles were characterized by UV-Visible spectroscopy, XRD, FT-IR, AFM, SEM, HR-TEM techniques. Based on the results the synthesized sulfur nanoparticles were proposed to have particle size in the range 2-15 nm. The synthesized nanoparticles were also evaluated for their capacity for reduction of hexavalent chromium into a less toxic state.

Trivedi et al [10] have reported green synthesis of copper nanoparticles using *Celastrus paniculatus* wild leaf extract. The synthesized copper nanoparticles were characterized using Uv-Visible spectroscopy, SEM, TEM, FT-IR and EDS techniques. Based on the results the synthesized nanoparticles were proposed to have particle size in the range 2-10 nm. The synthesized nanoparticles were also evaluated for their antifungal activities against F. oxysporum and for their photocatalytic activity.

Kulkarni and Kulkarni [11] have reported green synthesis of copper nanoparticles using *Ocimum sanctum* leaf extract. The synthesized copper nanoparticles were characterized using XRD and FT-IR techniques. Based on the results obtained it was proposed that the Ocimum sanctum leaf extract can reduce copper ions into copper nanoparticles within 8-10 minutes of reaction time.

Ananda Murthy et al [12] have reported synthesis of green copper nanoparticles using medicinal plant *Hagenia abyssinica (Brace) JF. Gmel.* leaf extract. The synthesized nanoparticles were characterized using UV-Visible, UV-DRS, FT-IR, XRD, SEM. EDXA, TEM, HRTEM and SAED techniques. The obtained results indicated that the nanoparticles were mixture of spherical, hexagonal, triangular, cylindrical and irregularly shaped copper particles. The average particle size of synthesized copper nanoparticles was proposed to be 34.76 nm. The synthesized nanoparticles were evaluated for their antibacterial activities against *E. coli, P. aeruginosa, S. aureus* and *B. Subtilis*.

N Motakef-Kazemi and M Yaqoubi [13] have reported green synthesis of bismuth oxide nanoparticles using Mentha Pulegium extract. The obtained product was structurally investigated using UV-Visible, FTIR, DLS, SEM, TEM, EDS and DRS methods. The product was also tested for its antibacterial activity against *Salmonella, E. coli* and *S. Aureus*.

JC Tarafdar et al [14] have reported microbial synthesis of phosphorous nanoparticles using tri calcium phosphate (Ca3P2O8) as precursor salt by employing soil fungi *Aspergillus tubingensis TFR-5*. The obtained phosphorous nanoparticles were characterized using DLS, TEM, HR-TEM, SAED analysis.

P Yugandhar et al [15] have reported synthesis of silica nanoparticles using an annual grass herb *Cynodon dactylon (L.)*. The obtained silica nanoparticles were structurally investigated

using UV-Visible, FTIR, DLS, XRD, SEM and TEM methods. The synthesized silica nanoparticles were tested against four human pathogenic bacterial strains B. Subitilis, S. aureus, P. aeruginosa and E. coli using disc diffusion method.

B Muneera et al [16] have reported synthesis of vanadium oxide nanorods (VONRs) using *Tinospora cordifolia* leaf extract. The synthesized VONRs were characterized using UV-Visible, FT-IR, XRD, EDX, SEM and TGA analysis methods. The obtained results indicated that the size of VONRs were in the range of 10-40 nm. The synthesized VONRs were also tested for their anti-angiogenic, DNA binding and cleaving activities.

Nishat et al [17] have reported synthesis of nickel oxide nanoparticles using *Toona ciliate, Ficus carica* and *Pinus roxburghii* plants extracts. The obtained nanoparticles were characterized using various methods such as UV-Visible, Powder XRD, FTIR, attenuated total reflectance spectroscopy, SEM, TEM, EDX, TGA and fluorescence spectroscopy. The used plant extracts and nickel oxide nanoparticles were evaluated for their antibacterial activities against *S. aureus, S. mutans, S. pyrogenes, C. xerosis, B. cereus, S. epidermis, E. coli, K. pneumonia and P. aeruginosa*.

M Balamurugan et al [18] have reported synthesis of iron oxide nanoparticles using *Eucalyptus Globulus* leaf extract. The synthesized iron oxide nanoparticles were structurally investigated using UV-Visible, XRD, TEM, SEM and FTIR methods.

N M Salem et al [19] have reported synthesis of nano- sized sulphur using *Melia azedarach* leaves aqueous extract, citric acid and its effect on plant growth. The obtained nanoparticles were characterized using various methods such as Powder XRD, FTIR, and SEM.

P Paralikar et al [20] have reported synthesis of Sulphur nanoparticles using four *medicinal plant* extracts and The synthesized nanoparticles were evaluated for their antibacterial activities against common pathogenic bacteria *Escherichia coli* and *Staphylococcus aureus* The synthesized sulphur nanoparticles were structurally investigated using UV-Visible, Zeta potential, XRD, TEM, SEM and FTIR methods.

Research gap(s)

Synthesis of nanoparticle using green methods are one of the hot topics in research in current situation. Many researchers around the world have published many research papers regarding synthesis of nanoparticles using biological resources. Still there are many resources including various fruit extracts and extracts of various plant parts which need to be explored.

Moreover precious metals like silver, gold along with copper are explored to a greater extent as compared to other metals and non-metals. Nanoparticles or oxides of all other metals and non-metals are also applicable in various fields of life. So nanoparticles of these metals and non-metals or their oxides can be synthesized using identified biological resources.

Importance of the study (International, national regional status, Scope of the study)

Today when whole scientific world is promoting to avoid use of hazardous chemicals and to promote green synthesis, biomediated synthesis of nanoparticles would be a praiseworthy and welcomed approach. This work will connect to different branches of chemistry such as analytical, inorganic, organic, medicinal and green chemistry.

Brief information about few important researchers working on the international, national and regional platform is presented here.

A. International status:

- Akl M. Awwad, Department of Nanotechnology and Materials, Royal Scientific Society, Amman, Jordan
- Hoda Jafarizadeh-Malmiri, Faculty of Chemical Engineering, Sahand University of Technology, Tabriz, East Azarbaijan 51335-1996 Iran
- Ghassan M Sulaiman, Biotechnology Division, Applied Science Department, University of Technology, Baghdad 10001, Iraq.
- Asim Umer, Department of Chemical Engineering, Muhammad Nawaz Sharif University of Engineering and Technology, Multan, Pakistan.
- D. V. Talapin, Department of Chemistry and James Franck Institute at the University of Chicago.
- Jano. Markhulia, Chief Research fellow at Vladimer Chavchanidze Institute of Cybernetics of the Georgian technical University, Tbilisi 0186, Georgia.
- M. Peruzzini, Associate Researcher at CNR-ICCOM, Italy.

- K. Adibkia, Biotechnology Research Centre and Faculty of Pharmacy, Tabriz University of Medical Sciences, Tabriz, Iran.

B. National status:

- T.N.V.K.V. Prasad, Nanotechnology Laboratory, Institute of Frontier Technology, Regional Agricultural Research Station, Acharya NG Ranga Agricultural University, Tirupathi, 517505, Andhra Pradesh, India.
- Amita Pathak, Department of Chemistry, Indian Institute of Technology Kharagpur, Kharagpur, West Bengal 721302, India.
- Sunja Devi K. R., Department of Chemistry, Christ University (Deemed to be University), Bangalore, India.
- Ravi Mani Tripathi, Amity Institute of Nanotechnology, Amity University Uttar Pradesh 201313, India
- Manoj Kumar, School of Bio-Medical Engineering and Department of Chemical Engineering, IIT (BHU), Varanasi-221005, India.
- Murali Sastri, Department of Chemical and Biological Engineering, Department of Materials Science and Engineering, Monash University, India
- Rizwan Hasan Khan, Interdisciplinary Biotechnology Unit, Aligarh Muslim University, Aligarh 202002, India.
- B. Devaprasad Raju, Department of Physics, Sri Venkateswara University, Tirupati, Andhra Pradesh-517502, India.
- Ummey Shameem, Department of Zoology, College of Science and Technology, Andhra University Visakhapatnam-530003, India.

C. Regional status:

- Mahendra Rai, Department of Biotechnology, Sant Gadge Baba Amaravati University, Amaravati 444602, Maharashtra, India.
- Suresh Ghotekar, Department of Chemistry, Sanjivani Arts, Commerce and Science College, Kopargaon 423 603, Savitribai Phule Pune University, Maharashtra, India.
- Balaprasad Ankamwar, Bio-Inspired Materials Research Laboratory, Department of Chemistry, Savitribai Phule Pune University (Formerly University of Pune), Ganeshkhind, Pune 411007, Maharashtra, India.

- Govind Kolekar, Department of Chemistry, Shivaji University, Kolhapur 416004, Maharashtra, India.
- Gotan Hiralal Jain, KKHA Arts, SMGL Commerce and SPHJ Science College Chandwad, Nashik 423101 Maharashtra, India.
- Arun Ingale, Department of Biotechnology, School of Life Sciences, Kavayitri Bahinabai Chaudhari North Maharashtra University Jalgaon-425001, India.

Objectives of the proposed research work

- Selection of suitable biological material for the proposed nanoparticle synthesis.
- Preparation of biological extract and testing for phytochemicals assisting the nanoparticle synthesis.
- Synthesis of metallic and non-metallic nanoparticles using biological extract prepared.
- Characterization of synthesized nanoparticles using various characterization techniques in terms of structural and morphological properties.
- Screening of synthesized nanoparticles for their biological activities and applications.

Hypothesis

- Synthesis of nanoparticles using green methods will be advantageous than the traditional physical or chemical synthesis.
- Green methods: An easy, simple, convenient and eco-friendly method for the synthesis of nanoparticles.

Research methodology

A review of literature published on nanoparticles has cleared the idea about the techniques used for synthesis of nanoparticles. There are several methods in the literature for the synthesis of metallic and non-metallic nanoparticles such as physical, chemical and biological methods which are reliable and easy to synthesize metallic and non-metallic nanoparticles. Owing to the limitations of physical and chemical methods we found biological methods or Green method is most suitable as they are easy, simple and convenient; they require less reaction time and are eco-friendly to the environment. Hence green methods will be used in present study.

Limitations of the work

The thorough reading of literature reveals that synthesis of metal oxide nanoparticles requires high temperature conditions in few cases. So lab facilities with high temperature arrangement will be essential, which can be troublesome. In this case, help from other schools in our university or reputed institutes in country can be taken.

Also challenges may be faced while exploring synthesized nanoparticles for various applications. The main focus will be to search for useful day to day life applications of synthesized nanoparticles.

Expected outcome

The successful completion of the research work will lead us to metallic and non-metallic nanoparticles which will be applicable in various fields of life.

Year wise plan of work and targets to be achieved

Although exact plan of work and exact time required cannot be predicted, the tentative year wise plan of work and targets to be achieved can be proposed as follows:

Sr. No.	Year	Plan of work	Targets
1	First year	a. Identification of biological sources such as plant parts, fruits etc. suitable for the proposed synthesis. b. Preparation of extract solution of these biological sources using proper solvent. c. Identification of metals and non-metals or organic entity.	a. At least 4-5 different biological sources should be identified. b. Extract solutions will be prepared using proper solvents and proper composition. c. At least 4-5 different metals or non-metals will be identified.
2	Second year	d. Screening of prepared biological extract for phytochemicals present using various chemical tests and Spectroscopic techniques. e. Synthesis of metallic and non-metallic nanoparticles f. Characterization of synthesized nanoparticle using various techniques	d. Phytochemicals present in the prepared biological extracts will be identified. e. At least 4-5 different types of nanoparticles will be synthesized. f. Synthesized nanoparticles will be characterized using various techniques
3	Third year	g. Analysis of obtained results h. Screening of synthesized nanoparticles	g. Analysis of obtained results will be done in the light of literature available.

		for their biological activities h. presenting the obtained results in the form of research publications & submission to reputed journals.	h. At least 2-3 biological activities will be screened h. Research papers will be written & submitted to suitable reputed journals.
4	Fourth year	i. Preparation and submission of final synopsis j. Arrangement of Pre-Ph.D. Viva k. Writing final Thesis and submission	i. Final synopsis will be submitted to the university. j. Pre-Ph.D. Viva exam will be arranged k. Final Thesis will be submitted.

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